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U.S. DEPARTMENT OF COMMERCE

NATIONAL BUREAU OF STANDARDS

# *Technical News*

BULLETIN

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U.S. DEPARTMENT OF COMMERCE

LUTHER H. HODGES, *Secretary*

NATIONAL BUREAU OF STANDARDS

A. V. ASTIN, *Director*

## NATIONAL BUREAU OF STANDARDS

# Technical News

## BULLETIN

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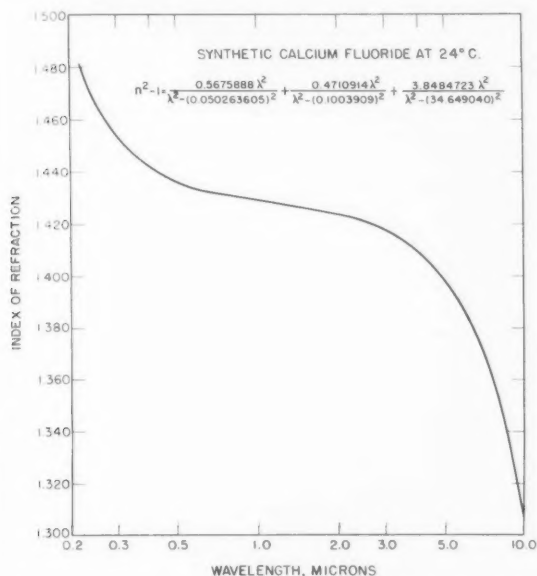
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**COVER:** Modified high-precision spectrometer used in recent National Bureau of Standards measurements of refractive indices of synthetic calcium fluoride. Refractive index values provide a basis for selecting optimum crystalline materials for the design of optical components and systems. Although synthetic calcium fluoride crystals are being produced in increasing numbers, comprehensive index values have not been published. The NBS study shows that the synthetic crystal is comparable in refractive properties to the natural crystal. (See story on facing page.)

# Optical Properties of SYNTHETIC CALCIUM FLUORIDE

FOR MANY years calcium fluoride has found important uses in the design of optical components and systems. Present applications in infrared and ultraviolet optical devices are becoming increasingly important, particularly in connection with military detection and guidance systems and space science research programs.



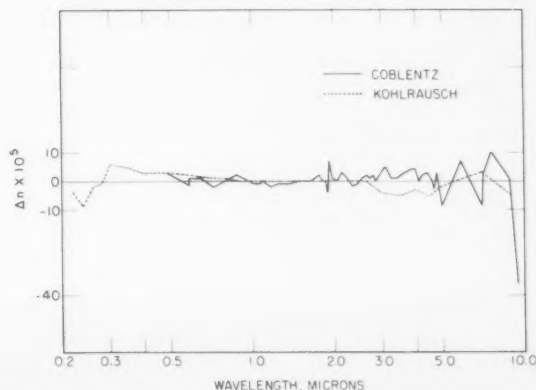
Dispersion curve obtained in a recent NBS study of the optical properties of synthetic calcium fluoride. Results show that the synthetic material, when properly synthesized, is comparable in refractive properties to natural crystals. The dispersion formula is valid for interpolation over the measured wavelength range, and gives values of refractive index accurate to five decimal places.

In selecting an optical material for a particular application, the optical designer must carefully consider the index of refraction of the material. Although synthetic calcium fluoride crystals have been available commercially for approximately 15 years, no refractive index values have been published for the synthetic material except in the visible region of the spectrum. For this reason, I. H. Maliton of the refractometry laboratory recently made refractive index measurements on both a synthetic and a natural crystal of calcium fluoride over a wide range of wavelengths.<sup>1</sup> Results indicate that synthetic crystals, when properly prepared, should be comparable in refractive properties to natural crystals.

The fact that the index data were obtained on a single instrument, under identical experimental conditions, and using identical procedures for different wavelength regions is considered an important feature of this work. Refractive indices of a synthetic prism of calcium fluoride were measured on a precision minimum-deviation spectrometer at controlled temperatures near 14 and 24 °C. Measurements were taken at 46 calibrated wavelengths over a range from 0.23μ in the ultraviolet to 9.7μ in the infrared. A natural prism was measured at 24 °C over the same spectral range. Observed data for the natural crystal were higher than those for the synthetic crystal by an average absolute value of only  $2.83 \times 10^{-5}$ ; thus the data for the two materials were in good agreement.

The dispersion curve, in which index values are plotted as a function of wavelength, largely dictates the design of the lenses and prisms that are incorporated into optical instruments. As the NBS measurements of refractive index were made only at specific calibrated wavelengths, some means of interpolation was necessary to determine indices at other wavelengths. Refractive index data observed for the synthetic crystal were fitted to a classical three-term Sellmeier dispersion equation containing six parameters. These constants were determined so as to give the best fit to the measured data; a high-speed digital computer was used in the determination.<sup>2</sup> Thus, using the dispersion equation with the calculated

A comparison of refractive index values of synthetic calcium fluoride with previously reported values for the natural crystal. The values for the synthetic crystal are represented by the line  $\Delta n = 0$ . It is apparent that the index values for the synthetic and natural crystal show good agreement.



parameters, intermediate values of the refractive index could be accurately interpolated to five decimal places.

In the present study, the dispersion equation was used to compute values of refractive index and dispersion at wavelengths separated by small regular intervals. Then these values were used to compute three quantities: the expected relative dispersion, contribution to chromatic aberration, and resolution of calcium fluoride as a function of wavelength. As these measures of dispersion involve only the index of refraction as a function of wavelength and are dependent only on the optical properties of the medium, they provide information suitable for the estimation of the optical performance of the material.

The index values obtained for the synthetic material were compared with data for natural crystals of calcium fluoride previously compiled at the Bureau by Coblenz in 1920.<sup>3</sup> The arithmetic mean of the absolute differences between the values was  $2.18 \times 10^{-5}$  for 57 wavelengths. The absolute average difference

between values obtained in the present study for the natural crystal and the synthetic crystal was  $2.83 \times 10^{-5}$ . Thus the values for the synthetic crystal show excellent agreement with those determined for natural crystals, both in the present study and in previous investigations. However, since no direct comparison has as yet been made between two or more samples of the synthetic calcium fluoride, further studies are needed to assess the extent of variability in refractive index between crystals of the synthetic fluoride.

<sup>1</sup> For further technical details, see A redetermination of some optical properties of calcium fluoride, by Irving H. Malitson, to be published in *Applied Optics* (Nov. 1963).

<sup>2</sup> Fitting refractive index data by least squares, by L. E. Sutton and O. N. Stavroudis, *J. Opt. Soc. Am.* **51**, 901-905 (1961).

<sup>3</sup> Transmission and refraction data on standard lens and prism material with special reference to infrared spectroradiometry, by W. W. Coblenz, *J. Opt. Soc. Am.* **4**, 432-447 (1920).



## TAYLOR APPOINTED ASSOCIATE DIRECTOR

LAURISTON S. TAYLOR has been named an Associate Director of NBS. In his new position, Dr. Taylor will be the principal staff advisor to the Director on NBS personnel, publications, and scientific support policies, and will continue to participate in the National Committee on Radiation Protection and Measurements, the International Commission on Radiological Units and Measurements, and the International Commission on Radiological Protection. He will also coordinate the Bureau's activities in radiation protection and measurement, represent the Department of Commerce on the Working Groups of the Federal Radiation Council, and act as NBS-AEC Coordinator.

Dr. Taylor, who was Chief of the NBS Radiation Physics Division, is a national leader in the field of radiation protection measures. Largely through the efforts of the National Committee on Radiation Protection, which Dr. Taylor has chaired since 1929, this country has established uniform radiation protection procedures for medical and industrial workers and the public. In directing the Bureau's radiation research program, he has obtained basic and theoretical data on the interactions of radiation with nuclei, atoms, and molecules, and has developed radiation sources and standards. He has also improved the means for detecting and measuring these radiations.

Prior to joining the Bureau's staff in 1927, Dr. Taylor received his education at Cornell University and worked briefly at the Bell Telephone Laboratories.

In 1941, he was designated Chief of the NBS X-ray Section, and concurrently, Chief of the Field Test Section of the Ordnance Development Division. From 1943 to 1946, he was Chief of the Operational Research Division of the 9th Air Force and later received a Bronze Star and the Medal of Freedom for this service. In 1948, he served as Chief of the Biophysics Branch of the AEC Division of Biology and Medicine and, a year later, was named Chief of the NBS Radiation Physics Laboratory.

He has made extensive contributions to the scientific literature in his fields and is coauthor of the book "Physical Foundations of Radiology." He has served on numerous national and international committees on radiation including the International Commission on Radiological Units and Measurements (Secretary 1934 to 1950, Chairman 1953 to the present) and the International Commission on Radiological Protection (since 1928; Secretary 1937 to 1950).

Dr. Taylor is the recipient of many honors and fellowships including the Sylvanus Thompson Medal (British Institute of Radiology), the Janeway Medal (American Radium Society), the Radiological Society of North America Gold Medal, the Department of Commerce Exceptional Service Award, and honorary membership in Der Deutschen Röntgengesellschaft.

# Infrared Structural Analysis of New Cesium Chloride Compound

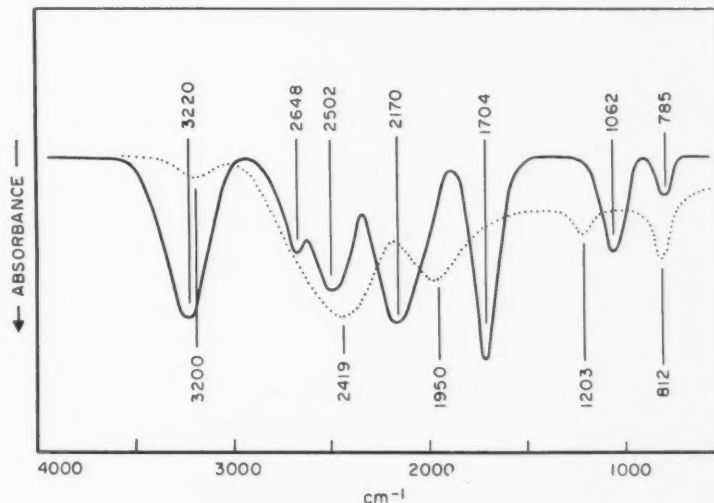
INFRARED spectroscopic analysis substantiates the hypothesis that a new compound obtained from aqueous cesium chloride solution saturated with hydrogen chloride is a mixed oxonium salt. The stabilization of the oxonium ion by cesium chloride at room temperature is considered remarkable.

Structural analysis of the new compound was a joint effort<sup>1</sup> by Arthur G. Maki of the Bureau and Robert West of the University of Wisconsin. The compound was first prepared in 1957 by Dr. West<sup>2</sup> in an effort to obtain the  $\text{HCl}_2^-$  ion. Since the chemical bonding in  $\text{HF}_2^-$  is not well understood, it was thought that study of another compound containing hydrogen and a halogen might lead to results that would help to clarify this type

and also prepared its deuterium analog in a similar manner from  $\text{DCl}$  in 97 percent  $\text{D}_2\text{O}$ . The spectrum of the deuterium compound should be the same as that of the basic hydrogen compound but displaced to a lower frequency; thus it would serve as a comparative check. Infrared spectral examination of the water liberated upon decomposition of the deuterium compound indicated that deuteration was more than 95 percent complete.

Infrared absorption spectra of both substances as mulls in mineral oil were recorded using a double-beam, grating-prism spectrometer that had been flushed with dry air. The overall agreement of the spectra with those of  $\text{H}_3\text{O}^+$  and  $\text{D}_3\text{O}^+$  is very good.

Infrared spectogram of a new cesium chloride compound  $4\text{CsCl} \cdot 3\text{H}_2\text{OCl}$  (solid line) and of its deuterium analog  $4\text{CsCl} \cdot 3\text{D}_2\text{OCl}$  as mulls in mineral oil (mineral oil bands have been deleted). The compound was first prepared at the University of Wisconsin. Because the results of various analyses differed, the compound was analyzed spectroscopically at NBS in an effort to determine its molecular structure and to verify the hypothesis that it contains the oxonium ion  $\text{OH}_3^+$ .  $\text{H}_3\text{O}^+\text{Cl}^-$  is normally unstable at room temperature; the cesium chloride in  $4\text{CsCl} \cdot 3\text{H}_2\text{OCl}$  must stabilize the oxonium ion.



of structure. Chlorine was chosen because it behaves more like fluorine than does either of the other halogens.

Partial analyses led Dr. West to believe the formula for the new compound was  $\text{CsHCl}_2$ . Recent, more nearly complete analyses have shown the correct formula to be  $\text{CsCl} \cdot \frac{3}{4}\text{HCl} \cdot \frac{3}{4}\text{H}_2\text{O}$  (or  $4\text{CsCl} \cdot 3\text{H}_2\text{OCl}$ ). Also,  $\text{CsHCl}_2$  has been prepared and found to be stable only at very low temperatures or high hydrogen chloride pressures. These findings imply that the compound obtained from aqueous solution does not contain the hydrogen dichloride ion  $\text{HCl}_2^-$ , but they do not reveal the actual nature of the compound.

As Dr. Maki and Dr. West surmised that the new compound  $\text{CsCl} \cdot \frac{3}{4}\text{HCl} \cdot \frac{3}{4}\text{H}_2\text{O}$  contained the pyramidal-form oxonium ion  $\text{OH}_3^+$ , they decided to analyze it spectroscopically in an effort to determine its structure and to verify their hypothesis. They prepared the compound, using the original procedure (see note 2),

However, the spectra are quite unlike those of typical ionic substances containing water of hydration. Bands characteristic of  $\text{HCl}$  and  $\text{DCl}$  are absent, as are bands at 1565 and 1180  $\text{cm}^{-1}$  that have been attributed to the  $\text{HCl}_2^-$  ion. Of the likely absorbing species, only the oxonium ion seems to be present; hence the spectral evidence suggests that the compound is actually  $4\text{CsCl} \cdot 3\text{H}_2\text{OCl}$ . This structure is entirely reasonable, for it is known that  $\text{HCl}$  in its solid monohydrate form transfers a proton to water, resulting in  $\text{H}_3\text{O}^+\text{Cl}^-$  (see note 2). At room temperature,  $\text{H}_3\text{O}^+\text{Cl}^-$  is unstable; the cesium chloride in  $4\text{CsCl} \cdot 3\text{H}_2\text{OCl}$  evidently exerts a stabilizing effect on the oxonium ion.

<sup>1</sup>For further details, see The nature of the compound obtained from aqueous cesium chloride solution and  $\text{HCl}$ , by Arthur G. Maki and Robert West, *Inorganic Chemistry*, (in press).

<sup>2</sup>R. West, *J. Am. Chem. Soc.* **79**, 4568 (1957).





C. S. Kopec, head of the new NBS gear metrology laboratory, adjusts the reference diameter of a master index plate so that it becomes concentric with the axis of rotation of a tooth index measuring instrument. Initial concentricity is denoted by the amplifier (left center). The two recorders show deviations in concentricity (left) and in tooth-to-tooth spacing (right) to within 5  $\mu$ in.

A GEAR metrology laboratory, recently set up at the Bureau, has a program under way to standardize gear measurement methods. Equipment for calibrating involute tooth forms and fine-pitch master gears has been acquired, and procedures are being devised for measuring large gears such as those used in radar scanners and naval vessels. The laboratory is under the supervision of I. H. Fullmer, Chief of the Bureau's engineering metrology laboratory, and it is headed by C. S. Kopec, who has had wide industrial experience in the design and evaluation of high-speed gears and gear drives.

The laboratory is partially the outgrowth of a series of measurement research conferences held at the request of the Aerospace Industries Association. At one of these sessions, systems design engineers and members of the gear industry reported that available techniques for determining the dimensions of gears and gear components were not sufficiently refined for the needs of modern technology. The Bureau has therefore undertaken the present program so that higher precision and accuracy in gear measurements may be obtained.

The new laboratory operates under controlled temperature and humidity conditions. Much of the equipment thus far installed is on loan from industry or from other Government agencies which are interested in improved gear calibrations. One device thus obtained, a unique recording master involute rolling instrument with a measurement precision of 5  $\mu$ in. is being used

Apparatus recently acquired to calibrate spur involute profile masters and master helical gears up to 8 in. in diameter. C. S. Kopec uses several gage blocks to measure the distance between the instrument's precision base and a 1.5-in. diam. involute master held by a set of precision centers. Instrumentation in foreground (counterclockwise): amplifier; autocollimator; straight line motion indicator.

# Gear Metrology Laboratory Established

to calibrate masters of involute profile approximately 1.5 in. in diameter. One of these masters was used recently to calibrate involute profile checking instruments in 16 gear laboratories throughout the country. The calibrations disclosed that improved techniques are needed to increase the precision capability of these instruments.

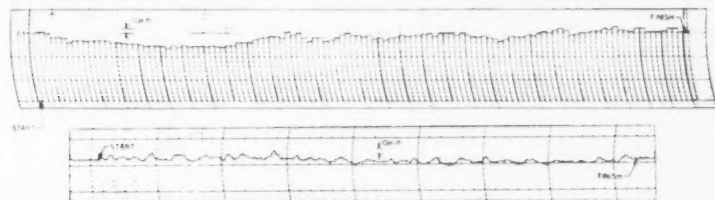
It is anticipated that a new involute measuring instrument, including a device for recording measurements at a 10,000 magnification, when completed to NBS specifications, can be adapted to the calibration of surface finish along the involute profile of spur and helical gears up to about 12 in. in diam. This equipment will eliminate the necessity of designing and fabricating the elaborate fixtures that would be required to adapt the master involute rolling instrument to the calibration of such gears.



*Right:* A master index plate in position for calibration. This plate will be used, in turn, to calibrate industrial instruments that check gear indexing and deviation from concentricity. The two master gears in the foreground can also be calibrated with the equipment shown.

The Bureau has recently purchased a tooth index measuring instrument, which will accommodate gear components from  $\frac{1}{8}$  to 8 in. in diameter, to measure the angular positions of individual gear teeth, master gears, index plates, and cutters to a precision of approximately 5  $\mu$ in. The instrument is a modified version of a prototype model which was recently evaluated by the Bureau. Extensive data obtained in the evaluation will be processed statistically to determine instrument accuracy.

The program for the calibration of large gears has been started with the acquisition of a 48-in. diam turntable which rotates concentrically to within 40  $\mu$ in. This table, mounted on a 10-ft long surface plate, will support gears up to 110 in. in diameter. However, equipment must be procured for tooth index, involute profile, helix angle, and surface finish measurements of such gears.

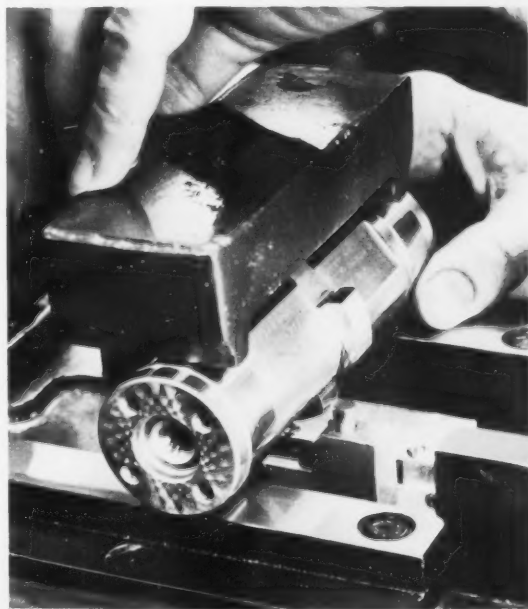
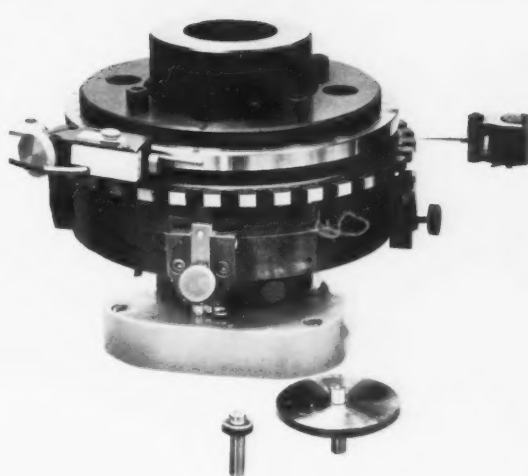


*Opposite:* The Bureau calibrates master index plates that are used in turn to calibrate the instruments employed by industry in checking gear indexing and eccentricity of gear diameter to the axis of rotation. These charts are typical of those obtained in the NBS calibrations. The upper chart shows linear deviation from the true index angle of each tooth on a master plate; the lower chart shows concentricity deviation.

A set of precision bench centers, consisting of two  $\frac{3}{32}$ -in. diam tungsten carbide balls, head and tail stock, and a 30-in. precision base, has recently been acquired for the calibration of helix or lead angle on spur involute masters and master helical gears up to 8 in. in diameter. The tail stock of the apparatus is vertically and horizontally adjustable so that the centers may be easily fitted to various sizes of gears. A device that will trace a straight line path to a 5- $\mu$ in. precision has been ordered for use with the apparatus. When completely assembled, the instrument will also be employed periodically to check the accuracy of the master involute rolling instrument now on hand.

Additional equipment that has been acquired for the laboratory includes two comparison autocollimators, an opposed-head electronic indicator and amplifier which records dimensions to 2  $\mu$ in., optical flats, precision gage blocks for measuring length and angle, and two permanent magnet blocks. When the large-gear instruments and other devices are installed, good facilities will be available not only for standardizing gear measurement methods but for providing assistance to other standards laboratories engaged in gear metrology.

*Right:* A spur involute profile master is positioned relative to an indicator pointer (lower right center) on the unique involute rolling instrument now installed in the gear metrology laboratory. The indicator pointer follows the involute profile of the master as it is slowly rotated, and any deviations in the profile are recorded at an 8,000 magnification on an automatic recorder (not shown).



# RADIAL FLOW APPARATUS for Thermal Conductivity Measurements

THE INCREASING number of high-temperature applications encountered in modern technology, as in nuclear reactors, rocket engines, and nose cones, has increased the need for accurate thermal conductivity data at elevated temperatures. To meet this need, the NBS heat transfer laboratory conducts a program to provide both techniques of measurement and thermal conductivity standards which will be useful at high temperatures.

Among the several possible methods for measuring thermal conductivities at high temperatures, the radial-heat-flow-in-a-cylinder method has the particular advantage that inherently it greatly reduces the need for thermal guarding or accessory thermal insulation to restrict unwanted heat flow which can cause serious errors in results. This feature is important because, especially at high temperatures, the accessory thermal insulation that may be needed in other methods, such as one using longitudinal heat flow in a cylinder, may be as conductive as the specimen being measured.

Typically, a radial heat flow apparatus has a cylindrical heater located along the axis of a cylindrical test specimen of length sufficient to avoid significant errors due to end conditions. Near the mid-length of the cylinder, heat flows radially outward from the axial heater and creates an angularly uniform distribution of temperature in the specimen, the temperature decreasing with increasing radius. After steady temperature conditions are attained, temperatures in the specimen at two different accurately known radii are measured, preferably at two or more symmetrical angular positions at each radius. The thermal conductivity of the specimen is calculated from these data, and from the measured heat input per unit of length of the axial heater.

Recently, a mathematical analysis of the radial-flow arrangement was made which showed that the positions of the heater and the thermocouples at the inner radius could be interchanged with some advantages from the experimental point of view. An apparatus employing the inversion of positions for heat input

and temperature measurement was constructed and used for measurements of the thermal conductivities of some granular insulations to 1100 °C.

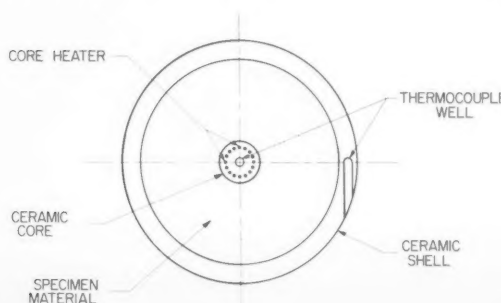
## Mathematical Analysis

Although inversion of the heating and temperature-measuring positions—if there is radial and angular symmetry—seemed valid intuitively, a mathematical analysis of radial heat flow systems with steady heating by continuous-line heat sources was made by B. A. Peavy<sup>1</sup> to investigate such systems in detail. His analysis included consideration of one and two concentric cylinders of different isotropic materials, heated by one or more continuous line heat sources parallel to the axis, and gave numerical solutions for multiple equal sources located with angular symmetry at a fixed radius. The method of solution for multiple unequal line sources at various radial and angular positions was also indicated. Values were given graphically for the cylinder length-to-radius ratio required to restrict, to within various limits, the error due to end heat-flow effects in cylinders of finite length. The analysis confirmed the validity of the inversion mentioned above, and provided a firm basis for making use of its experimental advantages.

Among the possibly important advantages of measuring the temperature along or near the cylindrical axis, stemming from the fact that this region is nearly isothermal, are the following: (a) the measuring thermocouple attains the temperature to be measured, and does not interfere with the pattern of radial heat flow, and thus may be made large, or be electrically insulated, or be clad for protection against chemical contamination or mechanical injury, and (b) the thermocouple can be moved longitudinally, without disturbing the pattern of radial heat flow, to measure the axial temperature at various longitudinal positions with the same thermocouple.

## Experimental Arrangement

An apparatus which illustrates this inversion of heater and thermocouple position was constructed by D. R. Flynn,<sup>2</sup> and used in the determination of the thermal conductivities of granular materials. The apparatus, shown in cross-section at left, consists essentially of a vertical ceramic heating core and a



Cross section of the radial-heat-flow apparatus used to make thermal conductivity measurements on powders to 1100 °C. In this apparatus the temperature is measured at the axis and heat is introduced at a known radius, which constitutes an inversion of the customary arrangement. Thus the inner thermocouple can be protected from contamination without interfering with its attainment of the temperature to be measured.

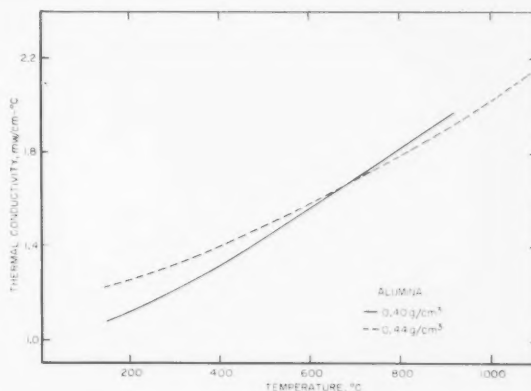


concentric ceramic shell, having a separation of 2.54 cm. The material under investigation is placed between these elements. The heating core, 46 cm long, has equally spaced holes extending through the entire length of the core, through which is threaded a continuous platinum-rhodium heating wire. A single thermocouple, which can be moved longitudinally to any selected position, is placed in the center of the core. End heat losses can be minimized by heating the ceramic end supports of the central core.

The outer ceramic shell is helically wound externally with heating wires, to enable heating it to a desired temperature. Here, too, end heat losses can be minimized by means of separate heaters wound at each end of the shell. Three thermocouples are inserted in tangential holes at the mid-plane of the outer shell, and the average of their indicated temperatures is used in the calculations. Additional thermocouples are placed in the shell, 15 cm above and below the mid-plane, to indicate its longitudinal temperature distribution.

Using this apparatus, the thermal conductivities of several granular materials were determined as a function of temperature over the range 100 to 1100 °C. Figure (right) shows the results obtained for finely divided alumina powder which had been produced at NBS by ignition of hydrated aluminum chloride.<sup>3</sup> The solid line represents the conductivity of a specimen having a test bulk density of 0.40 g/cm<sup>3</sup> and the dashed line represents that of a specimen of 0.44 g/cm<sup>3</sup> bulk density.

Thermal conductivity of powdered alumina as determined at NBS with the radial-flow apparatus. The solid curve is for alumina with a bulk density of 0.40 g/cm<sup>3</sup>, and the dashed curve for alumina with a bulk density of 0.44 g/cm<sup>3</sup>.



<sup>1</sup> Steady-state heat conduction in cylinders with multiple continuous line heat sources, by B. A. Peavy, *J. Res. NBS 67C (Eng. and Instr.)*, 119 (Apr.-June 1963).

<sup>2</sup> A radial-flow apparatus for determining the thermal conductivity of loose-fill insulations to high temperatures, by D. R. Flynn, *J. Res. NBS 67C (Eng. and Instr.)*, 129 (Apr.-June 1963).

<sup>3</sup> Development of a hydrochloric acid process for the production of alumina from clay, by J. I. Hoffman, R. F. Leslie, H. J. Caul, L. J. Clark, and J. D. Hoffman, *J. Res. NBS 37*, 409 (1946) RP1756.

## Visiting Scientists Selected for JILA Program

**TWELVE DISTINGUISHED** scientists from five countries will spend the 1963-64 academic year conducting research at the Joint Institute for Laboratory Astrophysics (JILA) on the University of Colorado campus.

The twelve have been selected from among many well-qualified applicants as participants in JILA's Visiting Scientist Program, according to Lewis M. Branscomb, JILA chairman.

JILA is a unique academic unit established in 1962 by NBS and the University of Colorado within the CU Departments of Physics and Astrophysics and Aeronautical Engineering. It is devoted to research and advanced training in areas vital to the expanding U.S. space program. The JILA program is of interest to several U.S. Government agencies and the Visiting Scientist Program has received support from the National Science Foundation.

### Coming from overseas:

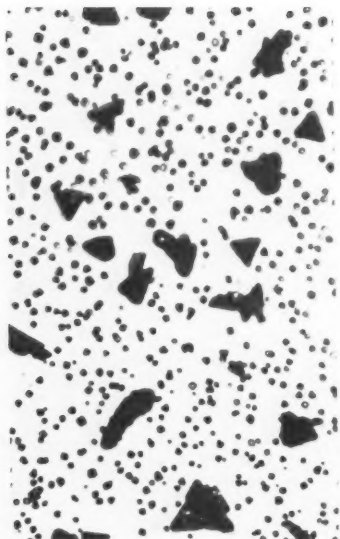
DR. PETER McWHIRTER of Culham Laboratory (Atomic Energy Research Establishment, England).  
 PROF. CHARLOTTE W. PECKER of the University of Paris.  
 DR. EIJIRO HIEI of Tokyo Astronomical Observatory.  
 PROF. R. GASPARD of Kossuth Lajos University, Hungary.  
 DR. DOUGLAS W. HEDDLE and DR. DAVID G. HUMMER of University College, London.  
 DR. ARTHUR E. KINGSTON of the University of Liverpool, England.  
 DR. KAZUO TAKAYANAGI of Saitama University, Japan.

### American visitors:

PROF. E. U. CONDON of Washington University (St. Louis).  
 DR. THOMAS F. O'MALLEY of New York University.  
 PROF. WILLIAM W. WILLMARTH of the University of Michigan.  
 DR. EDWARD C. ZIPF of Johns Hopkins University.

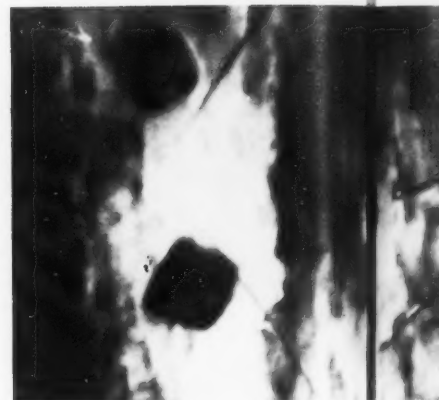
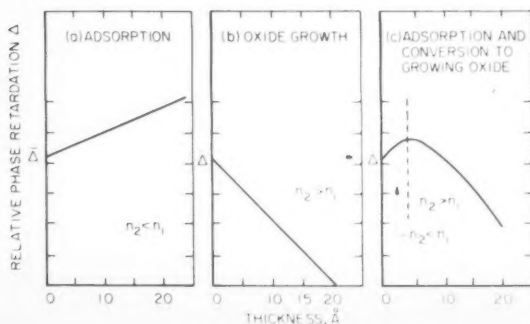
Most of the recipients will arrive in Boulder next August through October, and will spend a year at JILA working on problems of their own choosing in atomic physics, astrophysics, and aerodynamics.

# MECHANISM OF IRON PASSIVITY



Above: Passive film breakdown sites—some with a definite geometric pattern—found in a study of iron specimen surfaces. This specimen was immersed in a copper chloride solution to cause copper (small crystallites) to deposit at the cathodic breakdown sites, and gamma ferric oxyhydroxide (larger crystallites) at the anodic sites. ( $\times 990$ ).

Below: Three models derived theoretically to explain the mechanism of iron passivity illustrating how relative phase retardation  $\Delta$  would vary from the value  $\bar{\Delta}$  (found for a film-free surface) with film growth. In (a), the refractive index of an adsorbed film is less than that of the solution, and  $\Delta$  would rise above  $\bar{\Delta}$  with film growth. In (b), the refractive index of an oxide film is more than that of the solution, and  $\Delta$  would fall below  $\bar{\Delta}$  with film growth. In (c), the conversion of an adsorbed film to an oxide film would cause  $\Delta$  to rise above and then fall below the value found for  $\bar{\Delta}$  as the film grows.

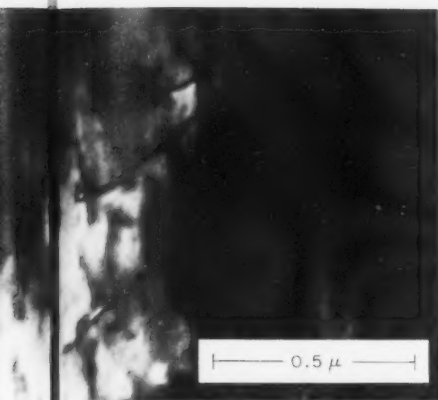


Electron micrograph of an iron foil specimen (discloses geometric figures) on the metal surface (location structure). This micrograph indicates the correlation phenomena since the surface intersections of the do not terminate at the breakdown sites.

**DIFFERENT THEORIES** have been reported on the composition of the passive film which makes iron surfaces resistant to corrosion. Some investigators have maintained that the film is an adsorbed oxygen monolayer. Others have held that it is a three-dimensional oxide film; and still others, that oxygen is first adsorbed and then converted to an oxide by the incorporation of a metal ion. However, evidence to support these theories has been inconclusive because investigators could not be sure they had initially film-free surfaces to study.

To help resolve the conflicting views and provide a better understanding of the iron passivity mechanism, Jerome Kruger of the metal reactions laboratory undertook a comprehensive study of the passivating process. He was able to observe the process while it was actually in progress through a combination of three experimental techniques—the use of an ultra-high vacuum system, ellipsometry, and simultaneous potential measurements. The results thus obtained confirm the oxide theory of passivity for films formed in inorganic passivating solutions.<sup>1</sup>

# N PASSIVITY STUDIED



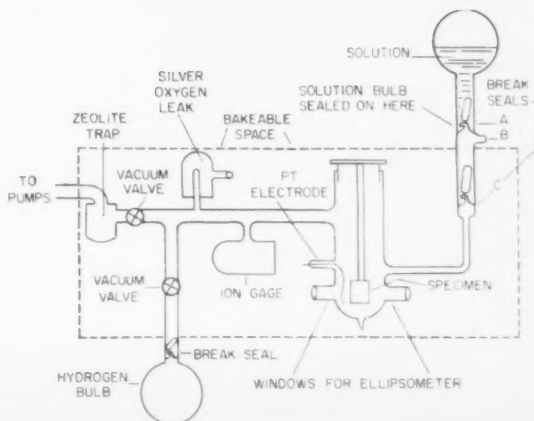
oil specimen discloses passive film breakdown sites (dark) surface dislocation defects (thin lines) in the metal. It indicates that correlation does not exist between the two intersections of the dislocations (the ends of the lines) and breakdown sites.

In the study, iron specimens were held at pressures of  $10^{-9}$  Torr in the vacuum system to obtain clean, well-characterized surfaces. An inorganic passivating solution was then introduced into the system to cover the specimens. Ellipsometric measurements of "film-free" surfaces and of film growth permitted calculations of film thicknesses as small as 5 Å. From electrochemical potential measurements of specimen surfaces during film growth, it was possible to determine film thicknesses that coincided with the onset of passivity.

In the ellipsometer, a polarized light beam is reflected from a metal surface in such a way that the ellipticity of the beam is altered after reflection. One of the important parameters characterizing the polarization state of the beam is known as relative phase retardation, and this parameter is used in calculating the thickness of thin films. For very thin films, the mean thickness is proportional to the difference between the phase retardation value found for a film-free surface,  $\Delta$ , and that found for a film-covered surface,  $\Delta$ .



*Above:* This pattern of passive film breakdown was found on the surface of an iron polycrystalline specimen. Copper precipitated from a copper sulfate solution at the breakdown sites shows that the density of the sites varies between grains. (X900). *Below:* Ultra-high vacuum apparatus used to study the mechanism of iron passivity by means of an ellipsometer while the passivating process is actually under way. Pressures of  $10^{-9}$  Torr obtained with the apparatus help in providing film-free specimen surfaces, so that the films that grow on the surfaces in a passivating solution may be accurately characterized. After the system is initially evacuated, hydrogen (lower left) is introduced to reduce any oxide film that may have formed on the specimen surface. The platinum electrode (center) measures the potential of the surface after the passivating solution has been introduced at the start of the passivating process.



## Theories Illustrated

Figure (p. 98, bottom) is helpful in interpreting the results obtained in the experiments. It shows how  $\Delta$  would vary with film growth (from the  $\bar{\Delta}$  shown on the ordinate), according to the three theoretical mechanisms of iron passivity.

The (a) curve, illustrating the first theory, is for an adsorbed film of a gas such as oxygen which has a refractive index lower than that of the solution ( $n_2 < n_1$ ). Hence, according to optical principles,  $\Delta$  would rise above  $\bar{\Delta}$  with film growth. The (b) curve illustrates the second theory: an oxide film, which has a refractive index greater than that of the solution ( $n_2 > n_1$ ), would cause  $\Delta$  to fall below  $\bar{\Delta}$  with film growth. The (c) curve, a combination of the first two curves, illustrates the third theory.

## Experimental Results

When an air-saturated nitrite solution was used in the first experiments,  $\Delta$  rose from an initially low value, temporarily approached the value that had been found for  $\bar{\Delta}$  (the specimen's film-free value), and then steadily decreased. All of the values found for  $\Delta$  were lower than  $\bar{\Delta}$ , indicating that an adsorbed film did not form. The initially low value found for  $\Delta$  suggested that an oxide film had instantaneously formed during the introduction of the solution. The temporary increase in  $\Delta$  which then occurred could have been caused by a slight film dissolution induced by the carbon dioxide present in the air-saturated solution.

To verify this assumption, a solution was then used to which only pure oxygen was added. The values found for  $\Delta$  in these experiments showed that no film dissolution occurred. The initially low value previously found for  $\Delta$  on the introduction of solution was again observed, and—instead of rising— $\Delta$  continued to decrease as the film continued to grow. Hence, the carbon dioxide present in the earlier work must have induced film dissolution.

In all these experiments, it appeared that the instantaneous film produced during the solution introduction interval could have been caused by interaction with oxygen and water vapor. So further experiments were undertaken in which a de-aerated, almost completely deoxygenated solution was used. In this part of the study, the values found for  $\Delta$  as the solution was introduced and shortly thereafter were extremely

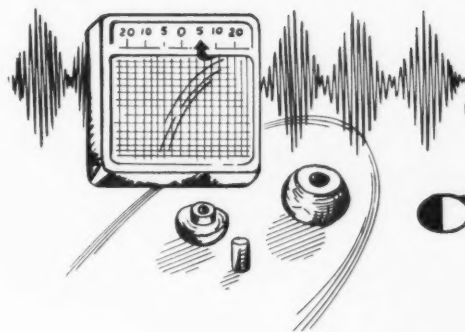
close to  $\bar{\Delta}$ , indicating little if any film formation. Extended exposure to the solution caused  $\Delta$  to decrease slowly, indicating the presence of an oxide film (as shown by the (b) curve of fig. on p. 98). Thus it appears that the oxygen in the air-saturated solution and the pure oxygen added in the earlier work had indeed caused instantaneous film formation, and that a passive film had formed in the present experiments after exposure to the inorganic passivating solution.

At the conclusion of the experiments, both optical microscopy and electron microscopy were used to investigate the influence of metal structure on the breakdown of passive film formation. The breakdown sites were pin-pointed by a technique that covered them with small crystallites. Two types of breakdown sites were found—cathodic sites covered by copper crystallites and anodic sites covered by gamma ferric oxyhydroxide. It was found that the number of sites per unit area varied with the crystallographic orientation of the iron surface, the [110] orientation always exhibiting the highest number. It was also found that no correlation existed between the breakdown sites and points where dislocation defects in the metal intersected the iron surfaces.

<sup>1</sup> For further technical details, see Optical studies of the formation and breakdown of passive films formed on iron single crystal surfaces in inorganic inhibitor solutions, by Jerome Kruger, *J. Electrochemical Soc.* 110, No. 6 (June 1963).



Jerome Kruger breaks one of the seals on an ultra-high vacuum system to permit a passivating solution (upper left) to enter and cover an iron specimen (hidden by the glass bellows, center) held inside the system. The growth of a passive film on the specimen surface will then be studied through windows on either side of the specimen by means of an ellipsometer (center and right foreground).



# STANDARDS AND CALIBRATION

## CORRECTIONS

### for use in low-temperature platinum resistance thermometry

**STEM-TYPE PLATINUM** resistance thermometers (prt's) usually are calibrated by the Bureau at the oxygen, triple, steam, and sulfur points. Thermometers so calibrated are then often used by other laboratories as references against which similar prt's are calibrated. Because of the potential risks involved in the use of liquid oxygen, many laboratories prefer to substitute a bath of liquid nitrogen, which has a temperature about 13 deg C lower, for their calibrations. Herein lies a difficulty.

The interpolation formula relating resistance to temperature on the International Practical Temperature Scale holds good only above the normal boiling point of oxygen (90.18 °K). Below this point, the IPTS is undefined and temperatures calculated with the IPTS formula are higher than the actual temperature being measured. Therefore, the NBS-55 scale is used from the oxygen point down to 10 °K. This scale is realized over its entire range solely with small capsule-type prt's. At the nitrogen point the value calculated with the IPTS formula will differ from the value on the NBS-55 scale by about 0.08 deg.

Platinum thermometers intended for use only *at the oxygen point and above* but which are calibrated by being compared at the nitrogen point with a standard prt calibrated only on the IPTS need not have this difference applied to their readings. This is because they are calibrated against a thermometer of similar material which has essentially the same behavior up through the oxygen point. However, if such thermometers are to be used below the oxygen point, down to about 50 °K, a table of corrections, such as that at right, should be consulted to bring the temperature within close agreement to NBS-55. Below 50 °K the characteristics of prt's differ sufficiently to make corrections for individual thermometers necessary.

*Corrections to computed temperature on NBS-55 scale<sup>a</sup>*

Temperature as computed using IPTS formula <sup>b</sup>	Correction to computed temp. to give values on NBS-55 scale <sup>c</sup>	Temperature as computed using IPTS formula <sup>b</sup>	Correction to computed temp. to give values on NBS-55 scale <sup>c</sup>
°K		°K	
90	-0.0000	70	-0.273
89	.0003	69	.311
88	.0008	68	.353
87	.0018	67	.399
86	.0036	66	.449
85	.0065	65	.505
84	.0107	64	.566
83	.0164	63	.634
82	.0236	62	.710
81	.0323	61	.794
80	.0428	60	.887
79	.0553	59	.990
78	.0698	58	1.103
77	.0865	57	1.229
76	.1054	56	1.367
75	.1266	55	1.519
74	.1503	54	1.688
73	.1766	53	1.874
72	.2055	52	2.080
71	.2376	51	2.309

<sup>a</sup> A similar table, based on experience at the National Research Council of Canada, has been prepared by R. L. Lovejoy.

<sup>b</sup> Degrees K are obtained from °C by adding 273.15.

<sup>c</sup> These values are based on measurements of thermometers which were manufactured in the United States and which meet the requirements and recommendations of the text of the IPTS. The corrections are not expected to be in error by more than 0.01 °C at the lowest temperature given when applied to temperatures determined with such thermometers.



# Controlled Temperature Oil Baths for Standard Cells

**SATURATED STANDARD** cells are widely used as voltage standards. In fact, groups of such cells are used as the references against which submitted cells are calibrated at NBS.

As the voltage generated by these cells is quite temperature dependent, a constant, known-temperature environment must be provided in which measurements can be made. Under the direction of Partick H. Lowrie, Jr.,<sup>1</sup> two oil baths, one maintained at 28 and the other at 35 °C, have been constructed and placed in operation for this purpose at the Bureau's Boulder (Colo.) Laboratories. These baths, which resemble similar installations at the NBS Washington Laboratories, have cyclic variations of less than  $\pm 0.001$  °C from a mean temperature which does not change by more than  $\pm 0.002$  °C per day. Thermal gradients, which severely affect the output of standard cells, are such that measurements with a platinum resistance thermometer show less than 0.001 °C difference over any 10-in. horizontal distance in the region of the cells.

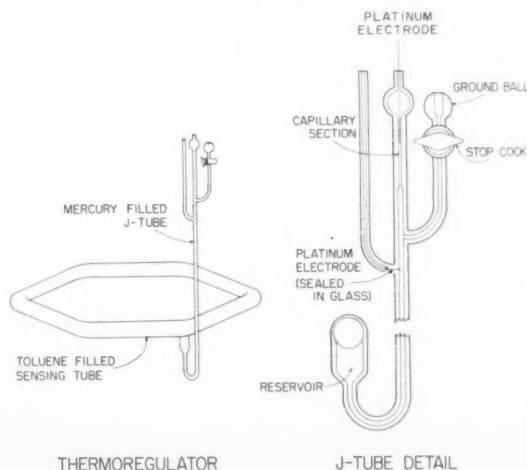
The baths, free-standing, self-contained units, are D-shaped (28 °C) and rectangular (35 °C). Within each bath are separate preheat, main, and drain tanks. Each bath is covered entirely with a hollow aluminum cover, 1 in. thick, which is filled with polystyrene foam, and crushed cork insulation is used between the main tank and the outer walls.

Racks of cells are immersed in oil in the preheat tank and brought close to the proper temperature before they are put in the main tank, thus reducing thermal instabilities which would occur if they were put into the main tank "cold." A separate heater and control system are used in the preheat tank. After cells are transferred to the main tank, they are allowed to stabilize for from 3 to 5 weeks before measurements are made. After calibration, the cells are placed in the drain tank while surface oil drains away, thus preventing spillage.



The main tanks are large enough to hold twelve groups of six cells each. During measurement the cells are contained in open-walled metal baskets, the baskets being suspended from the top to reduce danger of tipping. Oil is circulated within the main tank by means of a four-bladed stirrer rotated at 50 rpm. The stirrer has special blades, and the tank has an included baffle, to provide for the proper movement of oil over the heater and through the tank.

The main-tank heater, of open grid design to provide a short reaction time, is controlled by a Pyrex toluene-mercury thermoregulator. In this thermostat, expansion of the toluene (which has a coefficient of expansion about six times that of mercury) in a large sensing tube forces mercury up a capillary tube where contact is made between the electrodes controlling the heater. When contact is made a relay is tripped, reducing the current which is continuously supplied to the heater. Close control of the temperature is achieved through the use of an anticipator. This device, which consists of a small heater coil wound around the sensing tube of the thermoregulator, is supplied with current during the period when the higher current is supplied to the main heater. Because of the relatively greater effect in the regulator resulting from this additional heating, the regulator causes the current to the main



*Above:* Toluene-mercury filled thermoregulator for use in the controlled temperature oil baths. The expansion of toluene (which has a coefficient of expansion some six times that of mercury) forces mercury up the capillary to make contact with electrodes leading to the heater control circuit. *Opposite:* Two controlled temperature oil baths for use with saturated standard cells. The near bath is maintained at 35 and the far bath at 28 °C. These baths exhibit cyclic variations of no more than 0.001 °C from a mean which does not change by more than 0.002 °C daily.

heater to be reduced sooner than it would be normally, thus minimizing overshoot. No current flows in the anticipator when the main heater is on the low-heat phase.

As standard cells may be damaged by extremes in temperature, a special guard circuit is incorporated in each bath. These circuits, which make use of bimetallic regulators, take over control if the oil temperature changes by as much as 0.5 °C, and can maintain the temperature to within 0.05 °C. Also, should the line power to the baths fail, automatic switching to a small natural-gas-fueled generator would occur.

## Simplified Method for Sealing Sapphire Windows to Glass

A SIMPLIFIED method for sealing sapphire windows to glass tubes has been devised at the Bureau. Graded seals made by this technique are vacuum tight and are capable of withstanding temperatures as high as 400 °C and as low as -195 °C.

The need for a glass-sapphire seal grew out of NBS studies requiring a window in a xenon resonance lamp for observing ultraviolet spectral lines. Developed by Enrico Deleonibus and Gabriel Luther, the method will be useful to scientists studying electromagnetic radiation in the wavelength range of 0.1450 to 7  $\mu$ , the wavelengths transmitted by sapphire.

Bonding sapphire to glass is difficult because the difference in thermal expansion may cause either the glass or the sapphire to fracture when subjected to temperature changes. Some methods of sealing sapphire to glass require ovens and elaborate auxiliary equipment capable of producing and measuring precise temperatures. Other methods require expert glassblowers to combine and blend the glass holding the sapphire. The seal developed at the Bureau withstands thermal stresses and can be made with a minimum of specialized equipment.

In this method, commercially available single-crystal sapphires and glass tubes<sup>1</sup> are used. Cross-sectional cuts from the crystals result in disks approximately 0.33 mm thick and 13 to 32 mm in diameter, with the c axis perpendicular to the disk face.

Because of its wide laboratory use, a Pyrex glass

These baths have been in trouble-free operation for well over two years. The only difficulty has been a slow, slight rise in bath temperature resulting from contamination of the platinum electrodes of the thermoregulator due to oxidized mercury. However, cleaning every three or four months restores the temperature to within 0.005 °C of the proper value.

<sup>1</sup>For further details, see Controlled temperature oil baths for saturated standard cells, by Patrick H. Lowrie, Jr., NBS Tech. Note 141, 25¢. Order from the Superintendent of Documents, U.S. Government Printing Office, Washington 25, D.C.

tube is employed to start the seal. To this tube are joined four successive short lengths (about 5 mm) of glass tubing graded to approximate the expansion coefficient of Pyrex at one end and sapphire at the other. A mica sealing solder glass, ground to a fine powder, is then mixed with a small amount of amyl acetate to form a heavy paste. This paste is applied to the cut and cleaned end of the glass tube whose coefficient of expansion approximates that of sapphire. The sapphire window is placed over the end and the combined glassware is set vertically in a well-insulated oven and heated to 720 °C for 10 min. After cooling slowly, preferably overnight, the specimen is ready for testing and use.

This method was simplified further by employing commercially available Pyrex-to-soft-glass graded seals of the appropriate diameter. These seals consist of a continuous tube fabricated from Pyrex, six short lengths of sealing glass (graded by increasing expansion coefficients), and finally soft glass. The fourth grade from the Pyrex end is cut at about its midlength and the sapphire window is joined and finished as previously described. Thus, using this method, a minimum of glassblowing skill is required to effect the sapphire seal.

<sup>1</sup>The grades of glass employed are commercially available Pyrex-to-soft-glass series sealing glasses, and have expansion coefficients of  $40.5 \times 10^{-7}$ ,  $50 \times 10^{-7}$ ,  $60 \times 10^{-7}$ , and  $71 \times 10^{-7}$ , respectively.

## TNB Readership Survey

TO DETERMINE how well the *Technical News Bulletin* is meeting the needs of its readers, the Bureau recently surveyed a random segment of the TNB readership. Questionnaires were sent to approximately 10 percent of the subscription list and about 50 percent of the questionnaires were completed and returned. At the time of the survey, the paid circulation was approximately 5,000 in this country and about 500 in other countries.

The results of the survey have now been analyzed and are most encouraging. A large majority of the respondents felt that the TNB is an attractive, easy-to-read publication, written at the proper level for the audience to which it goes. No change in content or format seems necessary at this time.

A consistent theme of the reader comments was that the

TNB contains too little information on applied research and practical applications of research. However, many of those making such comments acknowledged that the National Bureau of Standards is primarily concerned with fundamental measurement problems rather than product or process development—a fact that is necessarily reflected in its publications.

The cooperation of those readers who participated in the survey is indeed appreciated. Their responses will be most helpful to the editorial staff in planning future issues. The editors will also welcome comments at any time from those readers who did not happen to fall within the statistical sample selected. Address reader comments to Office of Technical Information, National Bureau of Standards, Washington 25, D.C.



# STANDARD MATERIALS

## New Metal Standards, Primarily for Spectrochemical Analysis

Four groups of metal standard samples have recently been issued by the Bureau.<sup>1, 2</sup> Included in the new standards are four high-temperature alloys and ten copper-base alloys for calibrating optical emission and X-ray spectrochemical procedures. Two new Zircaloy-2 standards primarily designed for optical emission spectrochemical analysis have been made available; in addition, a revised certificate of analysis has been issued for one Zircaloy-2 and two zirconium metal standards which were made available earlier. A leaded steel standard suitable for both optical emission and X-ray spectroscopic analysis has also been issued.

The Bureau's standard materials program helps to provide a central basis for uniformity and accuracy in measuring. Over 325 materials have been certified for chemical composition, and nearly half of these have been prepared specifically for spectrochemical analysis. NBS standard materials are used for checking analytical methods and techniques, standardizing solutions for volumetric analysis, calibrating and standardizing spectrometers, colorimeters, pH meters, Geiger counters, and other scientific and industrial measuring devices.

### High-Temperature Alloys

Alloys having high yield strength at high temperatures are required in increasing amounts in modern technology. High-temperature alloys usually are complex in composition and therefore are analyzed mainly by spectrochemical and X-ray methods after suitable standard samples are available. To aid both production control and consumer acceptance analysis of the high-temperature alloys, the Bureau has issued four new analytical standards to supplement the eight already available. All are priced at \$25.00 per sample. The material for these standard samples was prepared in chill-cast form by the Allvac Metals Co., Monroe, N.C. The standards were checked for homogeneity by the Bureau. Chemical analyses were carried out by the Bureau and by The Carpenter Steel Co., Reading, Pa.; The International Nickel Co., Huntington, W. Va.; Ledoux and Co., Teaneck, N.J.; and, U.S. Steel Corp., Monroeville, Pa. The composition is given in table 1.

Table 1. High-temperature alloy standards

NBS No. <sup>a</sup> .....	1190	1203	1204	1205
Designation.....	Udimet 500	Inco 713-A	Inco 713-B	Inco 713-C
Element: <sup>b</sup>	%	%	%	%
C.....	(0.10)	(0.01)	(0.03)	(0.19)
Mn.....	.61	.31	.41	.29
Si.....	.22	.86	.56	.63
Cu.....	.093	.19	.12	.056
Ni.....	51.9	75.5	70.6	67.5
Co.....	19.1			
Fe.....	(0.6)	(1.4)	(3.1)	(1.55)
Cr.....	17.0	11.9	12.7	13.8
Mo.....	3.80	3.01	4.28	5.75
W.....	0.08	<0.01	0.028	0.019
Al.....	2.83	4.34	5.60	6.68
Ti.....	3.57	1.09	0.63	0.36
Zr.....	0.11	0.05	.12	.46
Nb (Cb).....	<0.01	1.00	1.31	1.95
Ta.....	<.01	0.34	0.46	0.67

<sup>a</sup> Size and metallurgical condition: Samples are approximately 1½ in. square and ¾ in. thick; they were chill-cast by a rapid unidirectional solidification technique.

<sup>b</sup> The standards also contain the elements boron, phosphorous, and sulfur which are expected to be certified at a later date.

<sup>c</sup> Values in parentheses are not certified, but are given for additional information on the composition.

### Copper-Base Alloys

The new copper-base alloy standards include five for cartridge brass (approximately 70% copper and 30% zinc) and five for red brass (approximately 85% copper and 15% zinc). In each of the two alloys, two standards are in wrought form and three are in chill-cast form. (A third wrought standard for the alloys could not be forged because of the high level of impurities, but the material may be extruded and issued later.) The material for each standard was melted and cast at the Naval Research Laboratory, Washington, D.C.

Table 2. Cartridge brass standards

NBS No. <sup>a</sup>	C1100 <sup>b</sup>	1101 <sup>c</sup>	C1101 <sup>c</sup>	1102 C1102
Element <sup>d</sup>	%	%	%	%
Cu.....	67.4 <sub>3</sub>	69.6 <sub>0</sub>	69.5 <sub>0</sub>	72.8 <sub>3</sub>
Zn.....	32.2 <sub>0</sub>	30.2 <sub>4</sub>	30.3 <sub>1</sub>	27.1 <sub>3</sub>
Pb.....	0.106	0.05	0.05	0.020
Fe.....	.072	.037	.037	.011
Sn.....	.055	.016	.016	.006
Ni.....	.052	.013	.013	.005
Mn.....	<sup>e</sup> (.003)	(.006)	(.006)	(.004)
P.....	(.01)	(.002)	(.002)	(.006)
Si.....	(.01)	(.005)	(.005)	(.002)

<sup>a</sup> Size and metallurgical condition: 1100 series are wrought samples 1½ in. in diameter, ¾ in. thick. C1100 series are chill-cast samples 1½ in. square, ¾ in. thick.

<sup>b</sup> The wrought material for this standard is not available.

<sup>c</sup> Small differences in the copper and zinc contents made necessary the separate analysis of the wrought and chill-cast material for this standard.

<sup>d</sup> Other purposely added elements having useful concentration ranges in the cartridge brass standards include: Aluminum, antimony, arsenic, beryllium, bismuth, cadmium, silver, and tellurium. Some of these are expected to be certified at a later date.

<sup>e</sup> Values in parentheses are not certified, but are given for information on the composition.

Table 3. Red brass standards

NBS No. <sup>a</sup> .....	C1109 <sup>b</sup>	1110 C1110	1111 C1111
Element:	%	%	%
Cu.....	82.2 <sub>2</sub>	84.5 <sub>0</sub>	87.1 <sub>1</sub>
Zn.....	17.4 <sub>3</sub>	15.2 <sub>0</sub>	12.8 <sub>1</sub>
Pb.....	0.075	0.033	0.013
Fe.....	.053	.033	.010
Sn.....	.10	.051	.019
Ni.....	.10	.053	.022
P.....	( <sup>c</sup> )	.....	.....

<sup>a</sup> Size and metallurgical condition: 1100 series are wrought samples 1½ in. in diameter, ¾ in. thick. C1100 series are chill-cast samples 1½ in. square, ¾ in. thick.

<sup>b</sup> The wrought material for this standard is not available. The high-impurity level precluded forging; however, some of the material was extruded and may be issued at a later date.

<sup>c</sup> Dashes indicate elements present but not certified.

After casting, the materials were tested for homogeneity by metallographic studies, optical emission, and chemical analyses at the Bureau; and by optical emission and chemical analyses conducted by a group in Committee E-2 of the American Society for Testing and Materials.<sup>3</sup> Chemical analyses were made on millings taken from the samples by the Bureau and by Anaconda American Brass Co., Waterbury, Conn.; Department of Mines and Technical Surveys, Ottawa, Canada; and the Mueller Brass Co., Port Huron, Mich. The price per sample of these materials is \$25.00. The composition is given in tables 2 and 3.

### Zirconium-Base Alloys

Zirconium-base alloys are becoming increasingly important because they have characteristics favorable for use in nuclear reactors. The Bureau has certified addi-

Table 4. Zirconium and Zircaloy-2 standards

[All concentrations are in parts per million unless otherwise designated]

NBS No. <sup>a</sup> ...	1210	1211	1213	1214	1215
Designation	Zirconium A	Zirconium B	Zircaloy-2D	Zircaloy-2E	Zircaloy-2F
Element: <sup>b</sup>					
Fe.....	0.25%	0.10%	0.06%	0.06%	0.25%
Cr.....	95	95	0.05%	0.10%	0.19%
Ni.....	8	26	0.01%	0.05%	0.09%
Sn.....	.....	.....	1.76%	1.60%	0.95%
Al.....	<sup>c</sup> (60)	(90)	(50)	.....	.....
B.....	(<0.25)	.....	.....	.....	.....
Cu.....	10	44	22	55	140
Mn.....	(5)	(7)	(6)	38	.....
Mo.....	.....	22	.....	30	(100)
Si.....	(30)	(100)	(30)	(120)	(350)
Ti.....	26	50	(33)	(50)	.....
W.....	(4)	(40)	.....	(40)	.....
U.....	1.8	2.3	2.0	45	9

<sup>a</sup> Size: Samples are disks 1½ in. in diameter and ¾ in. thick.

<sup>b</sup> Other elements also contained in the standards are: Cd, Co, Hf, Pb, V, P, Zn, Ga, Yb, C, O, and N.

<sup>c</sup> Values in parentheses are not certified, but are given for additional information on the composition.

tional elements in two zirconium and one Zircaloy-2 (a zirconium alloy containing about 1½% tin) standards previously issued,<sup>4</sup> and has issued two new Zircaloy-2 standards. Each standard contains more than twenty elements with about half of these certified at present. The average values of twelve of these elements are given on a provisional certificate of analysis issued with each sample. Since most elements present are at a contamination level of a few parts per million, the complexity of the homogeneity testing and analytical program resulted in a price of \$60.00 per sample—a price substantially higher than that charged for other spectroscopic standard samples. The material for the standards was provided by the U.S. Atomic Energy Commission. A single ingot for each standard was triple-arc melted and cast by the Albany (Oreg.) Station of the U.S. Bureau of Mines.

Laboratories cooperating in the analytical program included the National Bureau of Standards; Columbia-National Corp., Pensacola, Fla.; Reactive Metals, Inc., Ashtabula, Ohio; Westinghouse Electric Corp., Pittsburgh, Pa.; The Carborundum Co., Akron, N.Y.; General Electric Co., Schenectady, N.Y.; Oregon Metallurgical Corp., Albany, Oreg.; U.S. Atomic Energy Commission, New Brunswick, N.J.; U.S. Bureau of Mines, Albany, Oreg.; Wah Chang Corp., Albany, Oreg.; Metals and Control, Inc., Attleboro, Mass.; Westinghouse Electric Corp., Cheswick, Pa.; Babcock and Wilcox Co., Lynchburg, Va.; United Nuclear Corp., New Haven, Conn.; and the Allegheny Ludlum Steel Corp., Watervliet, N.Y. Table 4 shows the composition of the new standards and also the revised composition of the previous standards.



## Leaded Steel Standard

A leaded steel standard recently was issued by the Bureau for application in optical emission and X-ray spectrochemical analysis of this type of material. Lead is often added to steel where strength is not of prime importance but where freer and faster machining is desirable.

The solubility of lead in steel is extremely low and the lead appears as small, soft particles finely dispersed throughout the hard steel matrix. Although certain inhomogeneities, both linear and radial, were observed in the rods tested, suitable material has been selected for use as the NBS spectroscopic standard whereby the variation in either direction does not exceed 0.005 percent of lead.

Material for the standard was prepared by the American Steel and Wire Division of the U.S. Steel Corporation and was supplied to NBS in the form of 1 $\frac{1}{16}$  in. diam. rods. These rods were lathe cut to 1 $\frac{1}{4}$  in. in diameter and were subsequently cut in cross section to form disks  $\frac{3}{4}$  in. thick. The price per sample is \$15.00.

The samples were checked for homogeneity by the Bureau and were chemically analyzed by the Bureau and by the American Steel and Wire Division of U.S. Steel Corp., Duluth, Minn.; National Tube Division of U.S. Steel Corp., Lorain, Ohio; Jones and Laughlin Steel Corp., Pittsburgh, Pa.; and Jones and Laughlin Steel Corp., Cleveland, Ohio. The composition is shown in table 5.

Table 5. Leaded steel standard

NBS <sup>a</sup> No.	C	Mn	P	S	Si	Cu	Ni	Cr	Mo	Pb
1169.....	0.08	0.99	0.064	0.32	0.011	0.083	0.031	0.015	0.008	0.23

<sup>a</sup> Size: Samples are 1 $\frac{1}{4}$  in. in diameter and  $\frac{3}{4}$  in. thick.

<sup>b</sup> Inhomogeneity for lead was observed both from center to outside of the cross section, and from one end to the other longitudinally along the original rod. The observed variation was about 0.005 percent of lead in either direction.

<sup>c</sup> Figures in parentheses are not certified, but are given as added information on the composition.

<sup>1</sup> Standard samples issued by the Bureau are described in Standard Materials, NBS Miscellaneous Publication 241, available for 30 cents from the Superintendent of Documents, U.S. Government Printing Office, Washington 25, D.C. Up-to-date supplementary inserts, which are issued periodically, are available upon request directly from the Standard Sample Clerk, National Bureau of Standards.

<sup>2</sup> Orders for standard sample materials should be sent directly to the Standard Sample Clerk, National Bureau of Standards, Washington 25, D.C.

<sup>3</sup> Cooperating on ASTM Committee E-2, Subcommittee V, Task Group 3 are Bridgeport Brass Co., Bridgeport, Conn.; Chase Brass and Copper Co., Waterbury, Conn.; Mueller Brass Co., Port Huron, Mich.; Olin Mathieson Chemical Corp., New Haven, Conn.; Revere Copper and Brass, Inc., Rome, N.Y.; The Anaconda American Brass Co., Waterbury, Conn.; and Titan Metal Mfg., Bellefonte, Pa.

<sup>4</sup> Zirconium spectroscopic standard samples, NBS Tech. News Bull. 46, 48 (Mar. 1962).

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- CRPL Ionospheric Predictions* for August 1963. Three months in advance. Number 5, issued May 1963. 15 cents. Annual subscription: \$1.50; 50 cents additional for foreign mailing. Available on a 1-, 2-, or 3-year subscription basis.
- Journal of Research of the National Bureau of Standards*
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